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Torrefaction of oil palm mesocarp fiber and their effect on pelletizing

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ABSTRACT

The optimal condition for the torrefaction of oil palm mesocarp fiber was investigated by response surface methodology. Pelletizing properties of torrefied biomass were analyzed depending on torrefaction conditions. The elemental composition of torrefied biomass was influenced by the Severity Factor (SF) of torrefaction. The carbon content in the torrefied biomass increased from 48.02 to 54.83%, while the hydrogen and oxygen contents decreased with the SF. The calorific value of torrefied biomass ranged from 19.15 to 22.82 MJ kg^{-1}. This implied that the energy contained in the torrefied biomass increased by 5–19%, when compared with untreated biomass. The calorific value and weight loss in the biomass increased as the SF increased. The energy yield was high at a low SF, allowing for weight loss and calorific value. At a high SF, no pellets were made by torrefied biomass, or many defects were shown in the torrefied pellet, while the pellets were produced with good formation at a low SF.

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1. Introduction

Lignocellulosic biomass residues plays a major role in the production of sustainable energy, because it is abundant, relatively inexpensive, and often locally available. In recent years, growing attention worldwide has focused on the use of lignocellulosic biomass residues as a feedstock to produce biofuel pellet, as an alternative to fossil fuel [1,2].

Malaysia is the largest exporter of palm oil in the international market. In the process of extraction of palm oil from palm fruit, a mesocarp fiber is generated as a residue, which is obtained at the nut and fiber separator. In practice this mesocarp fiber is burned in incinerators by palm oil mills, which not only creates environmental pollution problems in nearby localities, but also offers limited value to the industry [3]. The annual amount of mesocarp fiber generated by the oil palm industry is 10.3 million tonne [4]. Therefore, availability of mesocarp fiber is considered to be the best among biomass residues [5].

The torrefaction process has been used for high energy density of lignocellulosic biomass. Torrefaction is a thermal

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pretreatment process where raw material is heated in an inert or nitrogen atmosphere, at a temperature of 200–300 °C [6]. This provides a hydrophobic condition for the biomass, due to the removal of the hydroxyl group during thermal treatment [7]. Therefore, the torrefied biomass provides suitable chemical and physical characteristics for long-distance transportation and long-term storage. Additionally, the advantages of torrefaction include a higher calorific value or energy density, lower moisture content, and better grinding [8]. During the torrefaction of biomass, most of the volatile compounds are removed from the biomass as vapors, and consequently results in a higher energy density. Many researchers have reported that torrefaction temperatures for lignocellulosic biomass range from 250 to 300 °C [2,6,8,9]. In addition, Chen et al. suggested that biomass subjected to light torrefaction (less than 1 h) is appropriate for producing solid fuels with a higher energy density, as compared to untreated biomass [10].

However, torrefaction of biomass accompanies weight loss, depending on the torrefaction condition. A high calorific value of the torrefied biomass is indicative of such weight loss during torrefaction. Therefore, torrefied biomass should be evaluated in terms of energy yield. Some studies have reported torrefied pellets/briquettes lead to poor quality [2,11]. It is implied that it is difficult to form torrefied pellet from torrefied biomass, under the same procedures as making regular pellets from untreated biomass.

In this study, we investigated the optimal condition for torrefaction by response surface methodology, and then made torrefied biomass into pellets in a single pelleter test unit. The effect of torrefaction severity for pellet production was also investigated.

2. Material and methods

2.1. *Materials*

Mesocarp fiber of the oil palm was purchased from New Star Inc. (Gyeonggi-do, Korea). The biomass was collected at an oil palm plantation in Kelantan, Malaysia in 2011. They were dried to below 10% moisture content for safe outside storage, and were used in this study without grinding.

2.2. *Torrefaction process*

The torrefaction conditions were based on a 2^2 factorial design augmented with star design (four axial points) and three replicates in the central point (Table 1). The mesocarp fiber was dried at 105 °C for 24 h before torrefaction in order to remove the water remaining in the biomass. The dried mesocarp fiber (300 g) was placed in a batch reactor (designed by Drying Engineering Inc., Korea), which has a temperature controller, and which was sealed to increase the temperature. The mesocarp fiber was torrefied under nine different conditions, with stirring. Torrefaction was performed under anoxic condition to avoid oxidation and ignition. A nitrogen flow of 2 L min^{-1} was used as the inert carrier gas. After torrefaction, the heater was turned off, and the reactor was left to cool down to room temperature. Torrefied biomass was passed through five different sieves (0.425 mm, 1 mm, 2.8 mm, 4 mm, 6.35 mm), resulting in the accumulation of six differently-sized particle collections. A schematic diagram of the torrefaction reactor is shown in Fig. 1.

For the torrefaction of biomass, a severity factor (SF) was used to integrate the effects of reaction times and temperature into a single variable [12]. The SF used in this study is defined as

$$\text{SF} = \text{Log}\left[t \cdot \exp\left(\frac{T_\text{H} - T_\text{R}}{14.75}\right)\right] \tag{1}$$

where t is the reaction time of the torrefaction in minutes, T_H the reaction temperature in °C, and T_R the reference temperature, most often 100 °C.

2.3. *Elemental analysis and calorific value of biomass*

The moisture content of torrefied biomass was performed using the oven-dry method [4]. The gross calorific value based on dry weight at constant volume was determined. The samples were weighed, and ranged from 0.5 to 0.6 g; combustion was performed in a Parr 6400 automated isoperibol bomb calorimeter. The ash content was determined by burning the oven-dried sample (1 g) in a platinum crucible in a muffle furnace model at 575 ± 25 °C. The nitrogen, carbon and hydrogen content of the torrefied biomass were assessed via

Table 1 – The 2^2 factorial design with four axial points and three replicates in the central point matrix employed for two independent variables.

Sample no.	Variables		Coded levels		Severity factor (SF)
	Time (min), X_1	Temperature (°C), X_2	Time, x_1	Temperature, x_2	
1	50	250	0	0	6.12
2	50	250	0	0	6.12
3	50	250	0	0	6.12
4	70	270	1	1	6.85
5	70	230	1	−1	5.67
6	30	270	−1	1	6.48
7	30	230	−1	−1	5.30
8	80	250	1.4	0	6.32
9	50	280	0	1.4	7.00
10	20	250	−1.4	0	5.72
11	50	220	0	−1.4	5.23

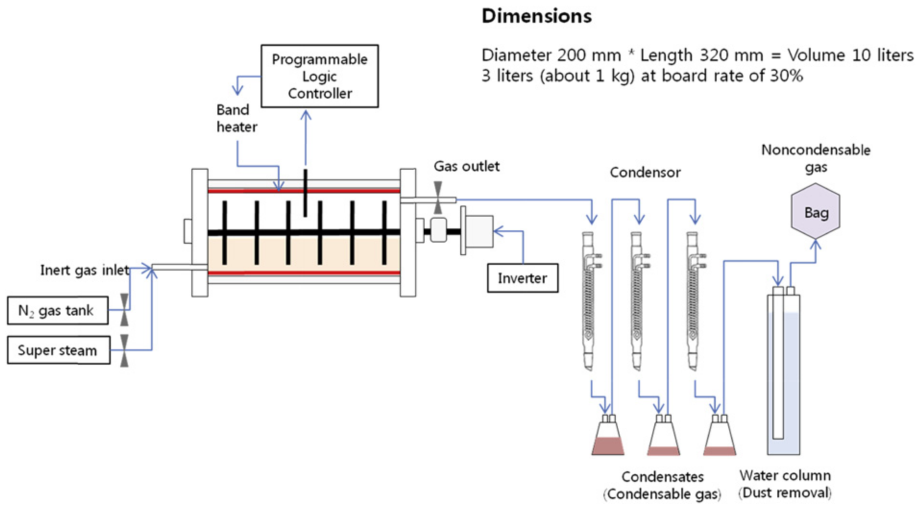
Dimensions
Diameter 200 mm * Length 320 mm = Volume 10 liters
3 liters (about 1 kg) at board rate of 30%
Programmable Logic Controller
Band heater
Gas outlet
Condensor
Noncondensable gas
Bag
Inverter
Inert gas inlet
N_2 gas tank
Super steam
Condensates (Condensable gas)
Water column (Dust removal)

Fig. 1 – The design of the torrefaction reactor used in this study.

direct combustion and thermal conductivity detection, by using an elemental analyzer (Thermo EA 1112A, USA) [8]. The sample was dropped into a hot furnace, and flushed with pure oxygen for very rapid combustion, forming combustion byproducts (CO_2, H_2O, NOx and N_2), and then passed through the furnace filter and thermoelectric cooler for subsequent collection in a ballast apparatus. The gases collected in the ballast were mixed, and a small aliquot was then used for further conversion of the gases. The remaining aliquot that was reduced was evaluated using the thermal conductivity cell for nitrogen, carbon and hydrogen content. The oxygen content was determined by an elemental analyzer (EA-1110, Thermo Quest, Italy). All experiments were performed according to the quality standard of the wood pellet [13], in triplicate, and a mean value was reported.

2.4. *Surface response analysis*

A 2^2 factorial design was used for optimizing the torrefaction of mesocarp fiber to improve its energy density for pellet production. Time (X_1, 20–80 min) and reaction temperature (X_2, 220–280 °C) were chosen as the independent variables. Calorific value, weight loss of biomass, and energy yield were used as the dependent output variables. Second-degree polynomials were calculated using Design-Expert Version 8.0.1 software (Stat-Ease Inc, USA) to estimate the response of the dependent variables.

Factors from each run were appropriately coded to real independent variables x_1 and x_2 for the statistical model, as shown in Table 1. The independent variables were [(condition of the run − condition at the central point)/step change of the factor]. Therefore, x_1 corresponded to [(time − 50)/20], and x_2 was [(temperature − 250)/20].

2.5. *Pelletizing*

The torrefied pellets were pelletized using a single pelleter (designed by Drying Engineering Inc., Korea). A schematic diagram of the single pelleter is shown in Fig. 2. The press consisted of a cylindrical die of 8 mm in diameter and 40 mm in length. The die was heated to 100 °C for pellet production. The compression rod speed was 127 mm min^{-1}. The end of the die was opened. Torrefied biomass was loaded stepwise in amount less than 0.25 g into the unit, and then pressed. The pressure was released after 5 s, the piston removed, and more torrefied biomass loaded and pressed until pellet formation occurred. The moisture contents of torrefied biomass were maintained at 10% before pelletizing.

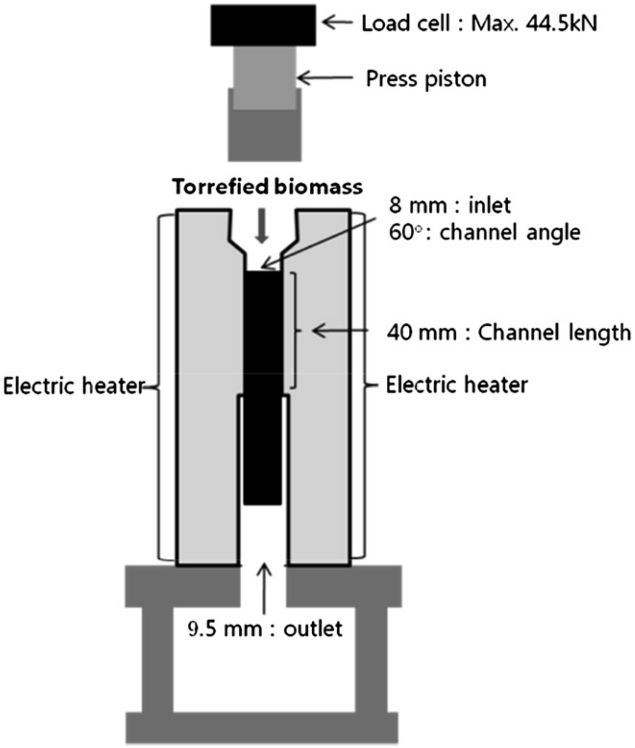

Fig. 2 – The design of the single pelleter used in this study.

3. Results and discussion

3.1. *Elemental composition of torrefied biomass*

The elemental component of untreated and torrefied biomass is shown in Table 2. The moisture content of torrefied biomass decreased from 7.84 to 0.98%, as the torrefaction severity increased. This behavior may be due to loss of hydroxyl groups from biomass during the torrefaction process. However, there is no trend for the ash content, depending on torrefaction severity. This result is very similar to that stated in the report by Uemura et al. [4]. The major ash compositions of oil palm wastes are K (1.6%), Ca (0.18%) and Mg (0.16%) [14]. Some of the potassium moves from the solid phase to the gas phase, depending on the torrefaction condition [15]. Therefore, torrefaction of mesocarp fiber provided unhomogeneity of ash content in the torrefied biomass.

Torrefaction of the mesocarp fiber resulted in a higher carbon, low oxygen and hydrogen content as the SF increased, resulting in decreased H/C and O/C ratios. This is due to the release of volatile compounds rich in hydrogen and oxygen, such as water and carbon dioxide. Torrefied biomass is believed to have a higher calorific value [6,16]. However, the nitrogen content remained almost constant.

3.2. *Response surface analysis of calorific value, weight loss and energy yield*

Calorific value and weight loss in the biomass over a range of SF values are shown in Table 3. The experimental calorific value and weight loss increased with SF. This result is in agreement with the report of Medic et al. [17]. These factors are major determinant in the energy density of biomass. In particular, the calorific value dramatically increased as the SF increased from 6.32 to 7.0 and when slowly increased until 6.32. This was due to removal of the moisture and volatile compounds remaining in the biomass, rather than to degradation of the major compounds of biomass, such as cellulose and lignin. This is similar to the change in carbon content (Table 2) in biomass during torrefaction.

Table 2 – Chemical compositions and elemental analysis of the torrefied biomass.

SF	Moisture content (%)	Ash (%)	C (%)	H (%)	O (%)	N (%)
5.23	2.68	8.39	49.90	5.86	20.64	2.25
5.30	2.64	10.08	50.36	5.37	19.69	2.29
5.67	2.54	10.00	50.46	5.22	19.80	2.20
5.72	2.33	9.52	50.56	5.44	19.17	2.27
6.12	2.66	9.35	51.95	5.20	18.37	2.22
6.12	2.70	9.53	51.73	5.23	18.19	2.36
6.12	2.49	9.44	51.41	5.03	18.71	2.40
6.32	1.76	11.75	52.40	5.05	17.90	2.19
6.48	2.19	10.67	54.83	5.08	16.31	2.40
6.85	1.31	9.96	54.51	4.95	16.75	2.20
7.00	0.98	10.26	57.08	4.85	14.63	2.37
Raw material	7.84	10.79	48.02	5.83	22.54	2.41

Table 3 – Calorific values and weight loss of biomass by torrefaction.

SF	Calorific value (MJ kg^{-1})	Weight loss (%)	Mass yield (%)	Energy yield (%)
5.23	20.11	10.89	89.11	93.59
5.30	20.26	11.18	88.82	93.97
5.67	20.44	11.83	88.17	94.11
5.72	20.50	21.37	78.63	84.17
6.12	20.47	21.22	78.78	84.21
6.12	20.72	21.69	78.31	84.73
6.12	20.53	21.34	78.66	84.32
6.32	20.70	26.79	73.21	79.15
6.48	21.97	29.13	70.87	81.32
6.85	21.85	36.71	63.29	72.20
7.00	22.82	39.22	60.78	72.44
Raw material	19.15	–	100	100

The calorific value ranged from 19.15 to 22.82 MJ kg^{-1}, depending on the torrefaction conditions. This implies that the energy content in the torrefied biomass increased by 5–19%, as compared to the untreated biomass. The increase in

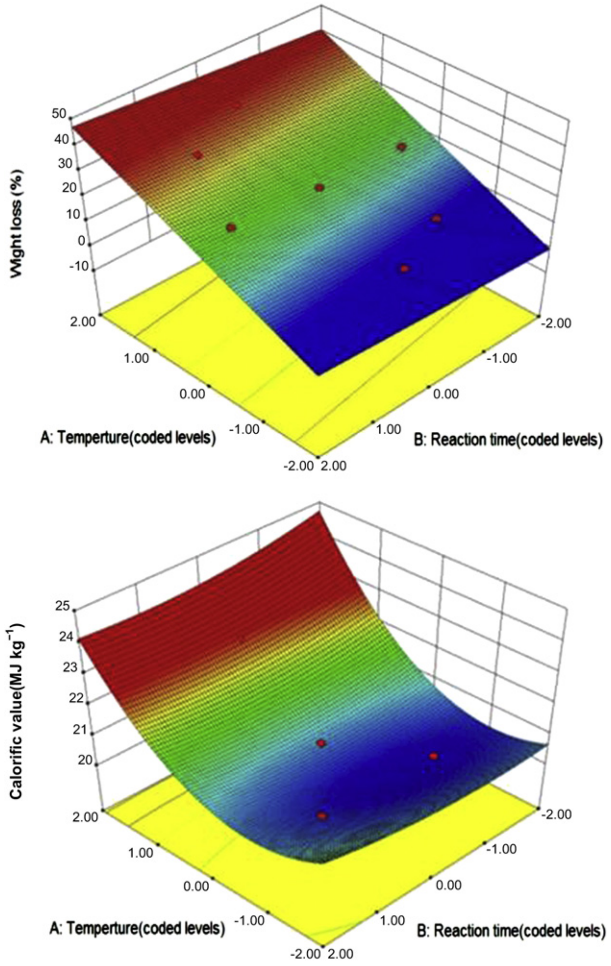

Fig. 3 – The response surface and contour plot of temperature vs. reaction time on the calorific value and weight loss during torrefaction.

Table 4 – Analysis of variance (ANOVA) for the adjusted model for the calorific value, weight loss and energy yield of biomass during torrefaction.

Source	Sum of squares	Degrees of freedom	Mean square	F-value	p-Value (Prob > F)
Calorific value					
Model	7.27	5	1.45	70.93	0.0001
Residual	0.10	5	0.02		
Lack of fit	0.07	3	0.02	1.34	0.4545
Purr error	0.03	2	0.02		
Corrected total	7.37	10			
Adjusted R-square: 0.9722					
Weight loss					
Model	856.54	2	428.27	87.17	<0.0001
Residual	39.31	8	4.91		
Lack of fit	34.37	6	5.73	2.32	0.3316
Purr error	4.94	2	2.47		
Corrected total	895.85	10			
Adjusted R-square: 0.9452					
Energy yield					
Model	358.10	2	179.05	15.50	0.0018
Residual	92.43	8	11.55		
Lack of fit	84.07	6	14.01	3.35	0.2477
Purr error	8.37	2	4.18		
Corrected total	450.54	10			
Adjusted R-square: 0.7435					

the calorific value of biomass during torrefaction was comparable with other similar studies for agricultural residues and wood chips [6,8,18]. The calorific value was affected more by the temperature than by the reaction time, as shown in Fig. 3. In particular, the calorific value significantly increased with a reaction temperature over 250 °C, regardless of the torrefaction time. The highest calorific value was 22.82 MJ kg^{-1} for the torrefied biomass at 280 °C for 50 min, corresponding to a weight loss of 39.22%. Based on the calorific value, the response surface determined the optimal torrefaction condition to yield the highest calorific value. The modeling results are provided in Fig. 3 and Table 4. A three-dimensional plot showed the predicted peak in calorific value at the optimum condition. Y_1 is the predicted calorific value (MJ kg^{-1}). The results of the analysis of variance (ANOVA) in equation (2) are presented in Table 4, where x_1 and x_2 denote reaction time and temperature. This model was significant at the 99% confidence level, and the p-values for temperature and reaction time were close to 0.

$$Y_1 = 20.573 + 0.007x_1 + 0.869x_2 - 0.075x_1x_2 + 0.075x_1^2 + 0.458x_2^2 \quad (2)$$

The weight loss of the biomass during torrefaction is shown in Fig. 3. The weight loss was calculated by equation (3). Reaction temperature has an impact on biomass weight loss during torrefaction. This might be due to the removal of water and volatile compounds present in the biomass as an initial reaction of the thermo-degradation of biomass. The temperature ranged from 220 to 280 °C in this study. In this range, most of the hemicelluloses, and some of the cellulose and lignin content in the biomass could be degraded during torrefaction. Weight loss highly depended on reaction

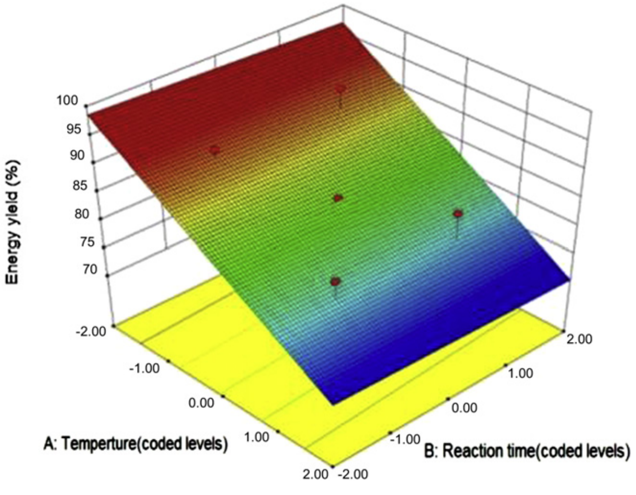

Fig. 4 – The response surface and contour plot of temperature vs. reaction time on the energy yield during torrefaction.

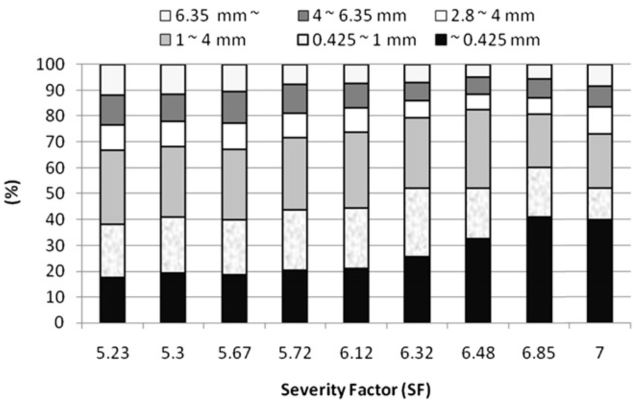

Fig. 5 – Profiles of particle size distribution from torrefied biomass at different torrefaction conditions.

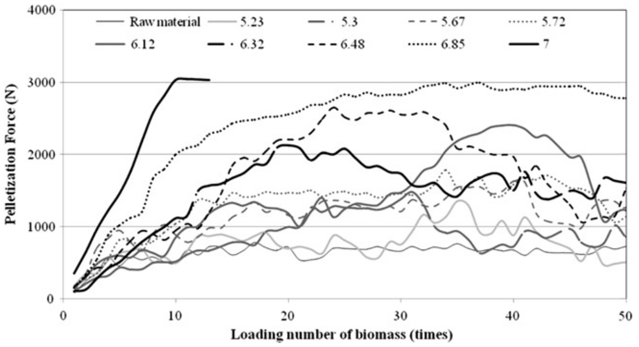

Fig. 6 – Pressure change curves during pelletization of raw material and torrefied biomass.

temperature, rather than time, during torrefaction. Table 4 shows the ANOVA of the quadratic model adjustment, where the total error was classified into lack-of fit and pure error. The F value estimated using the experimental data corresponded to the total residual and lack-of-fit values, respectively, and was lower than the tabular F value. This indicates that the models were significant in the region studied. For both models, the p-value was 0.0001, which shows that the models were strongly significant at a 99% confidence level. The model calculated for the weight loss of biomass (Y_2) is provided in equation (4).

$$\text{Weight loss}(\%) = \left(\frac{\text{mass of untreated biomass} - \text{mass of torrefied biomass}}{\text{mass of untreated biomass}}\right) \times 100 \tag{3}$$

$$Y_2 = 23.39 + 1.51x_1 + 10.24x_2 \tag{4}$$

The results of the energy yield in Table 3 range from 72.20 to 94.11%, depending on the torrefaction condition. They were calculated from the mass yield and calorific value using equation (5) and expressed as a percentage of the energy content of untreated dry biomass: $M_{\text{torrefied}}$ denotes dry mass of torrefied biomass; M_{initial}, dry mass of untreated biomass; $E_{\text{torrefied}}$, specific energy content of biomass after torrefaction; and E_{initial}, specific energy content of biomass before torrefaction.

$$\text{Energy yield}(\%) = \left(\frac{M_{\text{torrefied}}}{M_{\text{initial}}}\right) \times \left(\frac{E_{\text{torrefied}}}{E_{\text{initial}}}\right) \times 100 \tag{5}$$

The energy yield per untreated biomass indicates the total energy preserved in the torrefied biomass. It was highest when the difference between the weight loss and calorific value was high. Reaction temperature had the highest impact on the energy yield of torrefied biomass, while the effect of time was considerably lower. Below a SF of 5.72, the energy yield was maintained over 90%. However, the energy yield was very low at a high SF. This difference is mainly due to the poor mass yield of mesocarp fiber, while the calorific value increased to a similar extent by torrefaction. The energy yield was fitted to the response surface model provided in equation (6), in order to analyze the effect of the torrefaction factors on energy yield.

The present study using the technique of response surface methodology (RSM) enables accurate values of the torrefaction condition to be found for the maximum energy yield. Therefore, the optimal condition for the torrefaction of mesocarp fiber of oil palm is the red zone in Fig. 4, which could produce high energy density fuels, when the weight loss of biomass by torrefaction is considered.

$$Y_3 = 84.42 - 0.39x_1 - 6.68x_2 \tag{6}$$

3.3. *Pellet production*

The distribution of particle size particle weight percentage of torrefied biomass without grinding prior to feeding the single pelleter is shown in Fig. 5. Torrefied biomass at a high SF

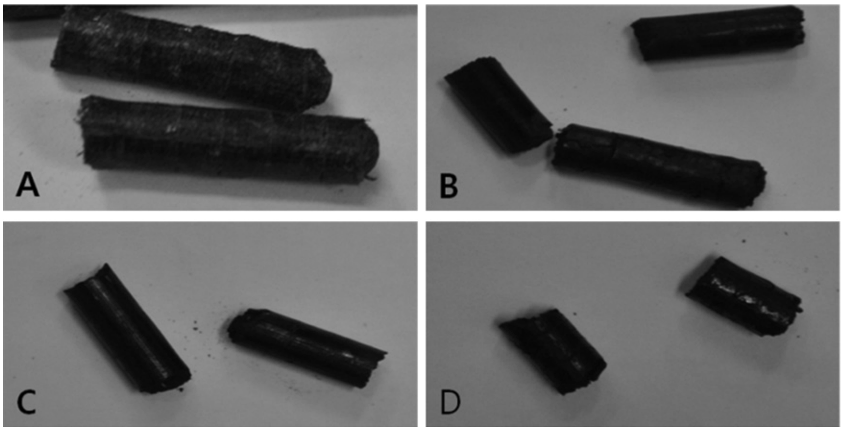

Fig. 7 – Pellets made from raw material and torrefied biomass (A: raw material, B: SF 5.23–5.67, C: SF 6.12–6.32, D: SF 6.48–6.85).

produced smaller particles than that torrefied at a lower SF. The torrefaction severity plays an important role in determining the size distribution. Similar trends have also been observed by others [8,9,19]. Overall, torrefied biomass produced relatively smaller particle size ranges compared to untreated biomass. This behavior directly saves the grinding energy consumption of torrefied biomass for pellet production.

According to the preliminary results, torrefied biomass was found to be difficult to form into pellets, under the same conditions as making control pellets. Therefore, a single pellet was produced by a die of 8 mm diameter and 40 mm length in this study. The die condition was appropriate to produce a single pellet over the range of the torrefaction conditions used in this study. Typical pressure change curves for compaction and extrusion of torrefied biomass in the single pelleter are shown in Fig. 6. The pressure increased, when comparing raw material and torrefied biomass. This implies lack of water and low hemicelluloses content in the torrefied biomass. Water and hemicelluloses play the parts of a binder and plasticizer. The hydroxyl group in water and hemicelluloses strengthen the bonds between individual particles in the pellets. In addition, they provide flexibility in the biomass [20,21].

The pellets were very different in quality (Fig. 7). No pellets could be made from torrefied biomass at a SF of 7.0, and even at a SF of 6.85 the pellet exhibits many defects. The pellet length increased with decreasing SF. At a high SF, the torrefied pellet has a short length and poor adhesion between the particles, while the torrefied pellet was produced with good formation at a low SF.

4. Conclusions

The optimal condition for the torrefaction of mesocarp fiber was investigated with respect to the reaction temperature and time. While the reaction temperature had a strong impact on the energy yield of torrefied biomass, the effect of reaction time was considerably lesser, under the torrefaction conditions used in this study. When the weight loss of biomass by torrefaction is considered, introducing low SF values for mesocarp fiber is appropriate for the torrefaction of biomass for the production of high energy density fuels. The torrefaction condition has a strong effect on the pelletization properties of the biomass. Overall, optimal torrefaction condition for pellet production was below SF of 6.37, which can be used to produce high energy yield and pellet with good formation.

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